THERMOPLASTIC RESIN COMPOSITION [Netsukasosei jushi soseibutsu]

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UNITED STATES PATENT AND TRADEMARK OFFICE Washington, D.C. January 2009

Translated by: FLS, Inc.

PUBLICATION COUNTRY	(19):	JP
DOCUMENT KIND	(12):	A
	(13):	PUBLISHED UNEXAMINED PATENT APPLICATION (Kokai)
PUBLICATION DATE	(43):	19910419 [WITHOUT GRANT]
PUBLICATION DATE	(45):	19910419 [WITH GRANT]
APPLICATION NUMBER	(21):	01234389
APPLICATION DATE	(22):	19890907
PRIORITY DATE	(32):	
ADDITION TO	(61):	
INTERNATIONAL CLASSIFICATION	(51) <b>:</b>	C08L 35/06, 51/04
DOMESTIC CLASSIFICATION	(52) <b>:</b>	
PRIORITY COUNTRY	(33):	
PRIORITY NUMBER	(31):	
PRIORITY DATE	(32):	
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TITLE	(54):	THERMOPLASTIC RESIN COMPOSITION

FOREIGN TITLE

[54A]: NETSUKASOSEI JUSHI SOSEIBUTSU

Name of the Invention
 Thermoplastic Resin Composition

### 2. Claim(s)

Thermoplastic resin composition comprising 10 - 80 wt.% rubberreinforced styrene resin (A), 0.5 - 20 wt.% epoxy-containing olefin
copolymer (B) made from an unsaturated epoxy compound and an olefin
and/or an ethylenically unsaturated compound, and 15 - 89.5 wt.%
maleimide copolymer (C) made from a maleimide compound and a compound
selected from an aromatic vinyl compound, a vinyl cyanide compound,
and an alkyl ester of an unsaturated carboxylic acid.

# 3. Detailed Explanation of the Invention [Industrial Field]

This invention pertains to a thermoplastic resin composition excellent in snap-fit property, heat resistance, impact resistance, and chemical resistance.

#### [Prior Art]

Rubber-reinforced styrene type resins as typified by ABS resin (acrylonitrile-diene type rubber-styrene polymer), AES resin (acrylonitrile-ethylene propylene type rubber-styrene polymer), ACS resin (acrylonitrile-chlorinated polyethylene-styrene polymer), and AAS resin (acrylonitrile-acrylic rubber-styrene polymer) are used for manufacturing automobiles, light electrical devices, general merchandizes, and the like for excellent impact resistance and

processibility thereof, wherein recent diversification and higher functionalities of these resins have created a need for higher heat resistance thereof.

Hence, there have been proposed and applied heat-resistant rubber-reinforced styrene type resins produced by composing  $\alpha$ -methyl styrene-acrylonitrile copolymer, anhydrous maleic acid-styrene copolymer, or N-phenyl maleimide-styrene-acrylonitrile copolymer with rubber-reinforced styrene type resins.

However, in the case of a component for a vehicle or a light electrical device, an assembly/installation projection is formed on the back face and needs to be temporarily deflected until fitted into and engages with a corresponding hole (groove), thereby creating a breakage problem (broken tip), where heat-resistant rubber-reinforced styrene resins are particularly susceptible to this problem.

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This hole-fitting property is generally called "snap-hit property" and evaluated based on the deflection bending ratio (the greater the deflection bending ratio, the better the property).

Although it is known that the snap-hit property can be improved by increasing the rubber amount contained in the rubber-reinforced styrene type resin or by increasing the molecular weight of styrene type copolymer which composes a continuous phase, such methods worsen the heat-resistance and processibility at the same time.

Furthermore, another problem with rubber-reinforced styrene type resins is low chemical resistance against grease, gasoline,

plasticizers, etc., and easily results in cracks by contacting with chemicals.

Although the chemical resistance is known to improve when an amount of vinyl cyanide compound content is increased, not only the impact resistance and processibility are worsened but also initial coloring (yellowing) becomes stronger; therefore, it is difficult to improve the snap-hit property and chemical resistance without sacrificing the primary characteristics of rubber-reinforced styrene type resin, particularly if the rubber-reinforced styrene type resin is heat resistant.

[Problems to be Solved by the Invention]

The developers of this invention thoroughly investigated these problems and discovered that a composition prepared by compositing a rubber-reinforced styrene type resin and two kinds of particular copolymers at a specific ratio could produce an excellent snap-hit property, impact resistance, heat resistance, and chemical resistance, and subsequently completed this invention.

Thereby, this invention provides a thermoplastic resin excellent in snap-fit property, heat resistance, impact resistance, and chemical resistance by composing 10 - 80 wt.% rubber-reinforced styrene type resin (A), 0.5 - 20 wt.% epoxy-containing olefin copolymer (B) made from an unsaturated epoxy compound and an olefin and/or an ethylenically unsaturated compound, and 15 - 89.5 wt.% maleimide copolymer (C) made from a maleimide compound and a compound

selected from an aromatic vinyl compound, a vinyl cyanide compound, and an alkyl ester of an unsaturated carboxylic acid.

Hereafter, the thermoplastic resin composition of this invention will be explained in detail.

The rubber-reinforced styrene type resin (A) in this invention is a graft copolymer obtained by polymerizing an aromatic vinyl type compound and another copolymerizable vinyl type compound if needed in the presence of a rubber-like polymer, or a mixture of said graft copolymer and an aromatic vinyl type copolymer obtained by polymerizing an aromatic vinyl type compound and another copolymerizable vinyl type compound on an as needed basis.

Examples of rubber-like polymer composing the rubber-reinforced styrene type resin (A) are diene-type rubber polymers, such as polybutadiene, butadiene-styrene copolymer, butadiene-acrylonitrile copolymer, etc., and non-diene type rubber-like polymers, such as ethylene-propylene copolymer, ethylene-propylene-unconjugated diene copolymer, acrylic rubber-like polymer, polyethylene chloride, etc., where one or more kinds of those materials may be used. These rubber-like polymers are produced by emulsification polymerization, solution polymerization, suspension polymerization, block polymerization, etc.

Although the particle diameter and gel content ratio of rubberlike polymer are not particularly restricted when the polymer is produced by emulsification polymerization, the average particle diameter is preferably 0.1 - 1  $\mu m$  and the gel content ratio is preferably 0 - 95%.

Examples of aromatic vinyl type compound are styrene,  $\alpha$ -methyl styrene, o-methyl styrene, m-methyl styrene, p-methyl styrene, t-butyl styrene,  $\alpha$ -methyl vinyl toluene, dimethyl styrene, chloro styrene, dichloro styrene, brome styrene, dibrome styrene, etc., where one or more kinds of those materials may be used. Particularly preferable materials are styrene and  $\alpha$ -methyl styrene.

Examples of other vinyl type compounds copolymerizable with an aromatic vinyl type compound are vinyl cyanide type compounds, such as acrylonitrile, methacrylonitrile, etc., unsaturated carboxylic alkyl ester, such as methyl acrylate, ethyl acrylate, butyl acrylate, 2-ethyl hexyl acrylate, methyl methacrylate, ethyl methacrylate, /3 propyl methacrylate, 2-ethyl hexyl methacrylate, etc., maleimide type compounds, such as maleimide, N-phenyl maleimide, N-methyl maleimide, N-cyclohexyl maleimide, etc., where one or more kinds of those materials may be used. Acrylonitrile, methyl methacrylate, and N-phenyl maleimide are particularly preferred among those.

As the graft polymerizing method, conventional emulsification polymerization, suspension polymerization, block polymerization, solution polymerization, or a combination of those is performed.

As the aromatic vinyl type compound composing the aromatic vinyl type polymer and another copolymerizable vinyl type compound, which are mixed with a graft copolymer, one or more kinds of materials may

be arbitrarily and respectively selected from the same group applied for producing a graft copolymer. Moreover, as for the polymerization method of these polymers, conventional emulsification polymerization, suspension polymerization, solution polymerization, block polymerization, or a combination of those is performed.

Although there is no restriction for the composition ratio of rubber-like polymer and compounds in the rubber-reinforced styrene type resin (A), the preferable ratio ranges are 20 - 80 wt.% rubber-like polymer and 80 - 20 wt.% compound. In addition, although no particular restriction exists for the composition ratio of aromatic vinyl type compound and another vinyl type compound among said compounds, the aromatic vinyl type compound is preferably 10 - 100 wt.%, particularly preferably 80 - 70 wt.%, and another vinyl type compound is preferably 90 - 0 wt.%, particularly preferably 70 - 30 wt.%.

The epoxy-containing olefin copolymer (B) in this invention is a copolymer consisting of an unsaturated epoxy compound and olefin, or a copolymer of those materials and an ethylene type unsaturated compound. The composition ratio of epoxy-containing olefin copolymer is not particularly restricted, however, the amount of unsaturated epoxy compound is preferably 0.05 - 95 wt.%.

The unsaturated epoxy compound is a compound containing an unsaturated group copolymerizable with an olefin and an ethylene type unsaturated compound and an epoxy group in a molecule.

Examples are unsaturated epoxy compounds, such as unsaturated glycidyl ester materials, unsaturated glycidyl ether materials, epoxy alkene materials, P-glycidyl styrene materials, etc. expressed as the following formulae (I), (II), and (III):

$$R - C - O - CH_2 - CH - CH_3$$
 (n)

(where R denotes a  $C_2$  -  $C_{18}$  hydrocarbon group containing an ethylene type unsaturated bond.)

(where R denotes a  $C_2$  -  $C_{18}$  hydrocarbon group containing an ethylene

type unsaturated bond; X denotes or .)

(where R denotes a  $C_2$  -  $C_{18}$  hydrocarbon group containing an ethylene type unsaturated bond; R' denotes a hydrogen or methyl group.)

Concrete examples are glycidyl acrylate, glycidyl methacrylate, itaconic acid glycidyl ester materials, butane carboxylic acid ester materials, aryl glycidyl ether, 2-methyl aryl glycidyl ether, styrene-P-glycidyl ether, 3,4-epoxy butane, 3,4-epoxy-3-methyl-1-butane, 3,4-epoxy-1-pentene, 3,4-epoxy-3-methyl pentene, 5,6-epoxy-1-hexane, vinyl cyclohexane monoxide, P-glycidyl styrene, etc., where

one or more kinds of those materials may be used. Glycidyl acrylate or glycidyl methacrylate are particularly preferred among those.

Examples of olefin are ethylene, propylene, butane, etc., where one or more kinds of those materials may be used. Ethylene and  $\frac{4}{2}$  propylene are particularly preferable among those materials.

Examples of ethylene type unsaturated compounds are vinyl ester materials containing  $C_2$  -  $C_6$  in a saturated carboxylic acid substance, acrylic acid and methacrylic acid ester materials containing  $C_1$  -  $C_8$  in a saturated alcohol substance, maleinic acid ester materials, halogenated vinyl materials, etc.

The amount of these ethylene type unsaturated compounds to be copolymerized with an unsaturated epoxy compound and olefin is no more than 50 wt.% of the total compound amount, where particularly preferable amount is 0.1 - 45 wt.%.

The epoxy-containing olefin copolymer (B) is produced by copolymerizing an unsaturated epoxy compound and olefin, and ethylene type unsaturated compound as needed basis or by graft-copolymerizing an unsaturated epoxy compound in the presence of olefin polymer or olefin and ethylene type unsaturated compound copolymer.

Preferable examples of epoxy-containing olefin copolymer (B) are copolymers prepared by grafting glycidyl methacrylate in the presence of an ethylene-glycidyl methacrylate copolymer, ethylene-vinyl acetate-glycidyl methacrylate copolymer, ethylene-methyl methacrylate-glycidyl methacrylate copolymer, and polyethylene,

polypropylene, poly-1-butene, poly-4-methyl pentene-1, and ethylenepropylene copolymer, ethylene-propylene-diene copolymer, etc.

To produce an epoxy-containing olefin copolymer (B), an emulsification polymerization method, suspension polymerization method, block polymerization method, solution polymerization method, or a combination of those is performed.

The maleimide type copolymer (C) in this invention is a copolymer consisting of a maleimide type compound (c-1) and at least one kind of compound (c-2) selected from aromatic vinyl compounds, vinyl cyanide compounds, and unsaturated carboxylic acid alkyl ester compounds.

The composition ratio of each compound composing the maleimide type copolymer (C) is not particularly restricted, however, particularly preferable amounts are 5 - 50 wt.% of maleimide type compound (c-1) and 95 - 50 wt.% of at least one kind of compound selected from a group consisting of aromatic vinyl compounds, vinyl cyanide compounds, and unsaturated carboxylic acid alkyl ester compounds.

Particularly preferable materials are a copolymer of maleimide type compound and aromatic vinyl compound and/or unsaturated carboxylic acid alkyl ester compound, a ternary copolymer of maleimide type compound, aromatic vinyl compound, and vinyl cyanate compound, a quaternary copolymer of maleimide type compound, aromatic vinyl compound, vinyl cyanide compound, and unsaturated carboxylic

alkyl ester compound. Moreover, the copolymer preferably has the viscosity (dimethyl formamide solution,  $3~9~{\rm ^{\circ}C}$ ) of 3.~8~~1.~3.

Although the maleimide type copolymer (C) can be obtained by directly copolymerizing the aforementioned compounds, it is also obtainable by imidizing the material with an amino compound after an anhydrous dicarboxylic compound, such as anhydrous maleic acid, is copolymerized with an aromatic vinyl compound or the like or during the copolymerization process.

Examples of maleimide type compound are, in addition to maleimide, N-aryl maleimide having an aryl group, such as phenyl, methyl phenyl, ethyl phenyl, chlorophenyl, etc., N-alkyl maleimide having an alkyl group, such as methyl, ethyl, etc., where one or more kinds of those materials may be used. Phenyl maleimide and O-chloro phenyl maleimide are particularly preferred among those.

Examples of aromatic vinyl compound, vinyl cyanide compound, and unsaturated carboxylic alkyl ester compound are the materials specified as examples for forming a rubber-reinforced styrene type resin (A). Styrene and  $\alpha$ -methyl styrene are particularly preferably used as an aromatic vinyl compound; acrylonitrile is preferably used as a vinyl cyanide compound; and methyl methacrylate is preferably used as an unsaturated carboxylic acid alkyl ester.

The thermoplastic resin composition of this invention consists of 10 - 80 wt.% rubber-reinforced styrene type resin (A), 1 - 50 wt.% epoxy-containing olefin copolymer (B), and 15 - 89 wt.% maleimide

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type copolymer (C), and the ratios other than these ranges will not result in an intended composition. Particularly preferable ratios are 15 - 70 wt.% rubber-reinforced styrene type resin (A), 3 - 40 wt.% epoxy-containing olefin copolymer (B), and 20 - 80 wt.% maleimide type copolymer (C).

Moreover, the rubber content in the composition (rubber content provided by a rubber-reinforced styrene type resin) should be 10 - 30 wt.% in view of the physical balance of composition.

The method for mixing the rubber-reinforced styrene type resin (A), epoxy-containing olefin copolymer (B), and maleimide type copolymer (C) is not particularly restricted, as these materials may be mixed in a latex state or in a form of powder, beads, pellets, etc. Moreover, the mixing sequence is not particularly limited and can be based on a method of mixing three components simultaneously or a method of premixing two components and adding the remaining component. As a melting-kneading method, conventional methods using a Banbury mixer, a roll, an extruder, etc. can be used.

Moreover, additives, such as antioxidizing agent, ultraviolet ray absorption agent, charge prevention agent, lubricant, dye, pigment, plasticizer, fire-retardant, mold-releasing agent, etc., may be composed as needed basis at the time of mixing. Moreover, an appropriate amount of thermoplastic resin, such as polyacetal, polycarbonate, polybutylene terephthalate, polyphenylene oxide,

polymethyl methacrylate, polyvinyl chloride, etc., can be added as well.

The following provide the concrete explanation of this invention based on examples and comparatives. Note that the parts and percentages are weight-based.

Reference Example 1: Production of rubber-reinforced styrene type resin (A):

<A-1>

After 100 parts of polybutadiene latex (average particle diameter = 0.4  $\mu$ , gel content ratio = 80%, solid = 50%),  $^{6.8}$  parts of potassium persulfate, and 100 parts of purified water were put in a nitrogen-substituted reaction vessel, the temperature was elevated to 65°C under stirring. Then, a monomer mixture solution consisting of 15 parts of acrylonitrile, 35 parts of styrene and 30 parts of an aqueous solution of emulsifier containing 0.2 parts of t-dodecyl mercaptan and 2 parts of disproportionate potassium rosinate were continuously and separately added for 4 hours, and then the polymer system was heated to 70°C and matured for 3 hours, thereby completing the polymerization, providing an ABS graft copolymer latex.

Then, after 1 part of Sumilizer BBM (registered trade name) and 2 parts of tris nonyl phenyl phosphite used as antioxidizing agents were added to 100 parts of (solid) ABS graft copolymer latex, the mixture was treated by a salting-out process with calcium chloride

and dehydrated/dried; as a result, a powder form of ABS graft copolymer was obtained (ABS-1).

#### <A-2>

After 0.3 parts of potassium persulfate and 120 parts of purified water were put in a nitrogen-substituted reaction vessel, the temperature was elevated to 65°C under stirring. Thereafter, a mixed monomer solution consisting of 30 parts of acrylonitrile, 70 parts of styrene, and 0.3 parts of t-dodecyl mercaptan and 30 parts of an aqueous solution of emulsifier containing 2 parts of disproportionate potassium rosinate were respectively continuously added for 4 hours; then, the polymer system was heated to 70°C and matured for 3 hours to complete a polymer, thereby providing an acrylonitrile-styrene copolymer latex.

Next, the material was treated by a salting-out process using calcium chloride and dehydrated/dried so as to produce a powder form of acrylonitrile-styrene copolymer.

After that, by mixing the ABS graft copolymer (A-1) and acrylonitrile-styrene copolymer, an ABS resin having 30% rubber content was obtained (ABS-2).

#### < A - 3 >

After 75 parts of EPDM (iodine value = 15.3, Mooney viscosity = 67, propylene content = 50%, diene component = ethylidene norbornene) were dissolved in 1,000 parts of n-hexane and 650 parts of ethylene dichloride, 75 parts of styrene, 25 parts of acrylonitrile, and 3

parts of benzoyl peroxide were added and polymerized at 67°C in an nitrogen atmosphere for 10 hours; as a result, an AES graft copolymer solution was obtained.

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After the polymer solution was put in a largely excessive methanol, an AES graft copolymer (AES) was obtained by separating/drying the deposited precipitant.

Reference Example 2: Production of epoxy-containing olefin copolymer (B):

Using an autoclave type polyethylene production device, an epoxy-containing olefin copolymer of the following composition was produced by a block polymerization method according to the polymerization condition of high pressure method polyethylene.

Ethylene-glycidyl methacrylate-vinyl acetate copolymer (composition ratio = 90-7-3).

< B - 2 >

Ethylene-glycidyl methacrylate copolymer (composition ratio = 90-10)

Reference Example 3: Production of maleimide type copolymer (C) <C-1>

After 70 parts of purified water, 0.02 parts of potassium persulfate, and 0.1 part of lauryl sodium sulfate were put in a reaction vessel, and the inside of reaction vessel was nitrogensubstituted, the materials were heated to 70°C under stirring. Then

these materials were heated to 75°C within 30 minutes while a solution consisting of 30 parts of N-phenyl maleimide, 20 parts of acrylonitrile, 50 parts of styrene, and 0.2 parts of t-dodecyl mercaptan and an aqueous solution consisting of 0.1 part of potassium persulfate, 1.0 part of lauryl sodium sulfate, and 50 parts of purified water were respectively continuously added. Upon completion of continuous addition of these solutions, the materials were matured at 75°C for 2 hours to complete the polymerization, thereby maleimide type copolymer latex was obtained.

Then, the material was treated by a salting-out process and dehydrated/dried; as a result, a powder form of maleimide type copolymer (C-1) was obtained.

< C - 2 >

A maleimide type copolymer (C-2) was obtained by the same method as described in C-1 except that 30 parts of N-phenyl maleimide was changed to 15 parts, and 50 parts of styrene was changed to 65 parts of  $\alpha$ -methyl styrene.

Reference Example 4: Production of styrene type copolymer

After 150 parts of purified water, 0.5 parts of potassium persulfate, and 2 parts of lauryl sodium sulfate were put in a reaction vessel, the temperature was elevated to  $70^{\circ}\text{C}$  under stirring. Then, a monomer mixture solution consisting of 30 parts of acrylonitrile, 70 parts of  $\alpha$ -methyl styrene, and 0.2 parts of todoecyl mercaptan was continuously added for 5 hours; next, the

polymer system was heated to  $75^{\circ}\text{C}$  and matured for 5 hours to complete the polymerization. As a result, an acrylonitrile- $\alpha$ -methyl styrene copolymer was obtained.

Thereafter, the material was treated by a salting-out process with calcium chloride and dehydrated/dried to provide a powder form of acrylonitrile- $\alpha$ -methyl styrene copolymer (D-1).

Examples and Comparatives:

The rubber-reinforced styrene type resin (A), an epoxycontaining olefin copolymer (B), and a maleimide type copolymer (C)
or a styrene type copolymer (D) obtained by the aforementioned
methods were mixed based on the compositions shown in Tables 1 - 3,
melting-mixed using a 40 mm biaxial extruder and made into particles.

The physical properties of obtained compositions were measured by the following method, and the results are shown in Tables 1 - 3.

Note that the test piece for measuring the physical properties was molded using a 3.5 oz injection-molder.

Snap-hit property: After a 1/8"x5"x1/2" test piece was placed on a bending test jig (span = 30 mm) supporting at both ends, a bending force was added at a bending speed of 20 mm/min., and the deflection bending ratio was calculated:

$$\frac{6d\Delta\ell}{L^2}\times100(\%)$$

d: Test piece thickness

 $\Delta \ell$ : Deflection amount at the breaking

# L: Span

Heat resistance: ASTM D-648

Impact resistance: ASTM D-256

Chemical resistance: ASTM -- Using an I type dumbbell, DOP (dioctyl phthalate) was coated over the surface thereof while a 1% prescribed deflection was applied to the specimen; then, the specimen was left unattended in an 80°C oven for a week, taken out, and rated based on the visible appearance.

Cracks Existed:  $\Delta$ 

" None: O

Broken specimen: X

Table 1 Table 2

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Key a) Example; b) Comparative; c) -Composition-(%); d) Rubber-reinforced styrene type resin (A); e) Olefin containing an epoxy group (B); f) Maleimide type copolymer (C); g) Styrene type copolymer (D); h) -Physical property-; i) Snap-hit property; j) Heat resistance; k) Impact resistance; l) Chemical resistance--Crack existence/non-existence or breakage; m) Did not break; n) Commercial general ABS (rubber content = 12%); o) Commercial heat resistant ABS (rubber content = 12%) copolymer containing  $\alpha$ -methyl styrene-acrylonitrile

	Table 3							
	<b>强 8 凝</b>		a)	b)				
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c)	-KK- (%)							
d)	ゴム強化ステレン系機器例			,				
e)	AES	25	S 0	40	100	36	40	
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h)	物性							
i)	ロスナップヒット性*男 *	>40	>40	>40	>4.6	15	28	
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Key a) Example; b) Comparative; c) -Composition-(%); d) Rubber-reinforced styrene type resin (A); e) Epoxy-containing olefin (B); f) Maleimide type copolymer (C); g) Styrene type copolymer (D); h) - Physical property-; i) Snap-hit property; j) Heat resistance; k) Impact resistance; l) Chemical resistance--Existence/non-existence ( $\Delta$ , 0) of crack or breakage (x); m) Did not break; p) Regular commercial AES resin (rubber content = 20%)

## [Effect of the Invention]

This invention provides the title composition excellent in snapfit property, heat resistance, impact resistance, and chemical resistance compared with the conventional (heat resistant) rubberreinforced styrene type resins.